Lawrence Berkeley National Lab

TI 900 TriboIndenter Nanoindenter User Manual

Location: Building 62

v. 1.7

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Jake Sadie & Rohini Sankaran

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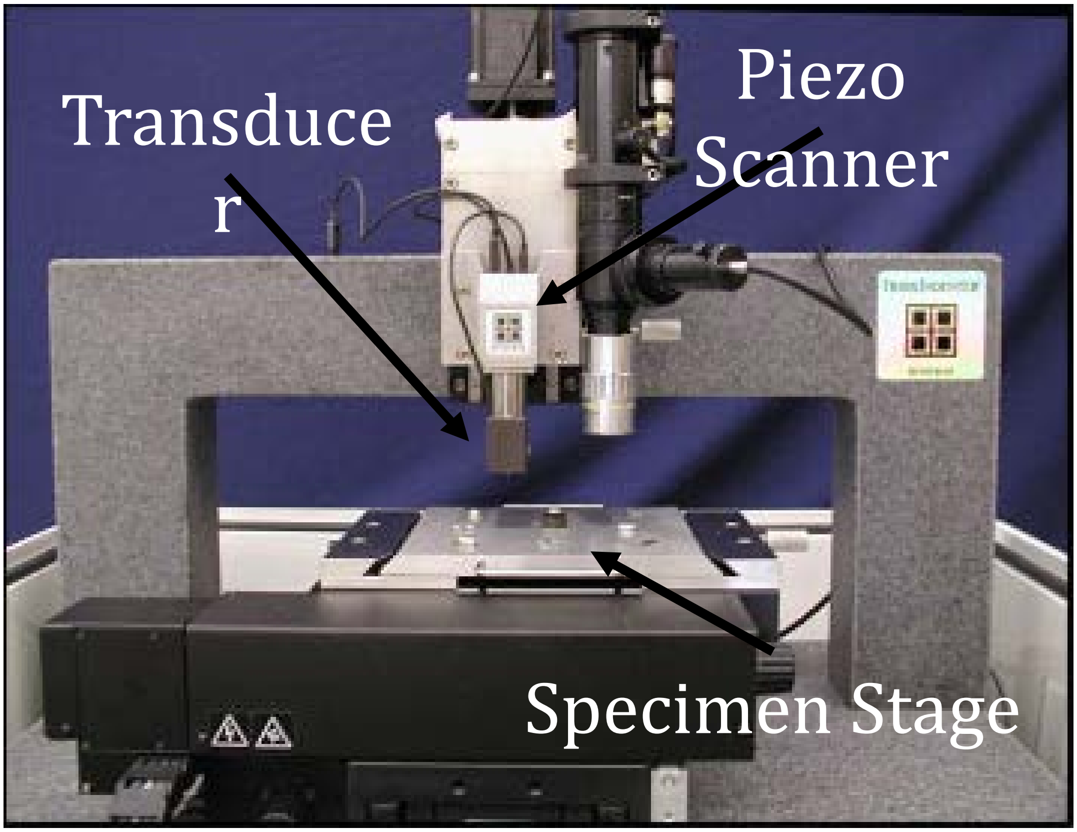
I. Nanoindenter Components

The instructions included in this manual is for work performed on the Hysitron TI 900 Model Nanoindenter. Below is a description of each of the main components of the nanoindenter setup.



a. Tip

The most commonly used tip in nanoindentation setups, and the tip used in this system, is a diamond Berkovich tip which has a three-sided pyramidal shape. The tip is mounted to the bottom of the transducer, which is mounted onto the piezo scanner.

b. Piezo Scanner

The function of the piezo scanner, called the TriboScanner, is to provide very fine resolution X, Y, and Z movements in fine approach of the sample. It is comprised of multiple piezoelectric cylinders which expand/contract depending on the voltage delivered and allow for deflections in the intended direction. Nominally, these deflections should be linear with applied voltage. The transducer is mounted at the base of the TriboScanner.

Operating Ranges:

X, Y Range:	0 – 60 µm
Z Range:	0 – 3 µm
Operating Voltage:	+/- 185 V

c. Transducer

The transducer is the heart of the nanoindenter system. Its purpose is to both apply and measure forces or displacements. The transducer converts shifts in capacitance measurements into displacements when the tip is in contact with the sample. Applied voltages (up to 600 V) inside the transducer also result in applied forces between the tip and sample.

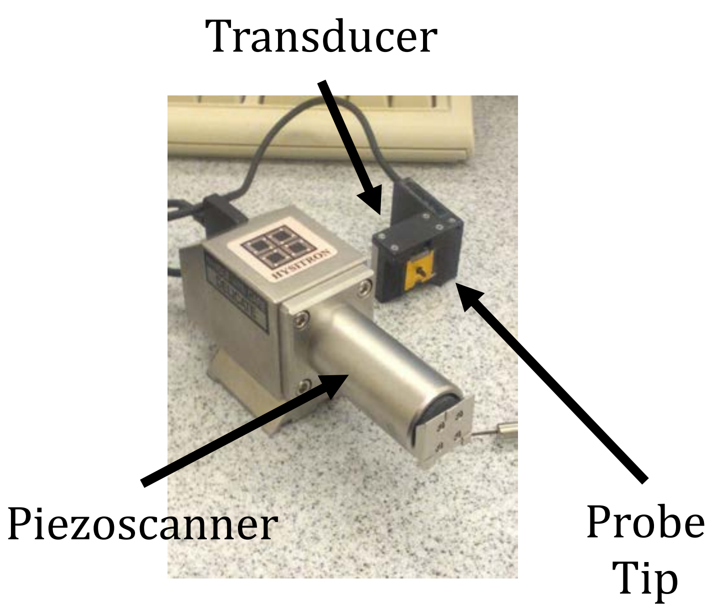
Two transducer types are available: 1D and 2D. The 1D transducer measures only vertical forces/displacements, while the 2D transducer measures vertical and lateral forces/displacements. The standard transducer available is the 1D transducer. Please refer to the TriboIndenter Manual for details on the 2D transducer.

Each transducer in use has a characteristic electrostatic force constant (ESF), which relates the required electrostatic applied force to the subsequent tip displacement. Over large displacement distances, the relationship between force and displacement is nonlinear, therefore it is critical to know and calibrate the electrostatic force constant before all experiments using an "*Air Indent*" *calibration* test.

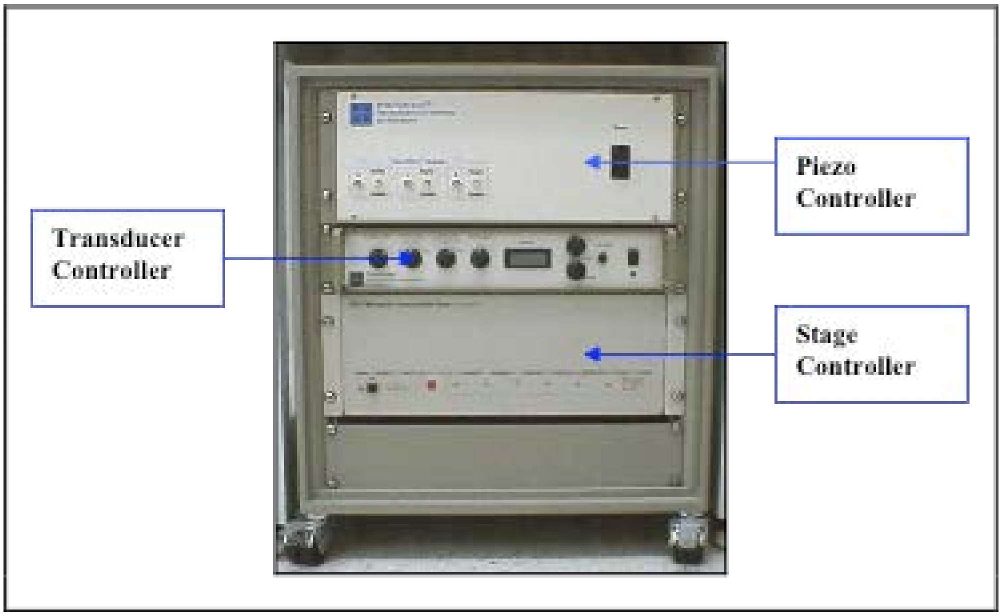
In addition to understanding the electrostatic force constant for the transducer, it is important to calibrate the system for potential noise caused by thermal drift in the transducer. This is done by holding the tip at a nominally constant applied force value after first contacting the sample and measuring the shift in measured displacement. The parameters for this *Drift Monitoring and Analysis* can be set in the load-control function setup.

Operating Ranges:

Maximum Force:	30 mN
Load Resolution:	1 nN
Maximum Displacement:	5 µm
Displacement Resolution:	0.04 nm



d. Electronics Rack



Below the scanning system is a rack with three electronic components required to control the system operation. They are (from top to bottom) the piezo controller, transducer controller, and stage controller.

Piezo controller: The piezo controller manages all signals delivered to the piezo scanner to position the tip near the surface of the sample. Most importantly, the piezo controller provides feedback for the piezo scanner to hold the tip on the surface with a constant force both before and after tests. **The piezo controller should always be turned off when testing is complete, as leaving it on without software control may lead to unstable controller states. Make sure the computer is on *BEFORE* turning on the piezo controller.**

Transducer controller: The transducer controller is used to adjust offsets, gain levels, and filtering for measured/applied signals. You will need to adjust the offset regularly. You will set the gain for the both the microscope feedback and displacement level (depending on the test you want to apply) to adjust the piezo feedback signal and z-axis data signal, respectively. **The transducer controller should always be turned off when testing is complete.**

Stage controller: The stage controller drives the X, Y, and Z stepper motors to position the sample beneath the tip. **The stage controller should always be left on.**

e. Other components

The following list details the other components of the nanoindenter system.

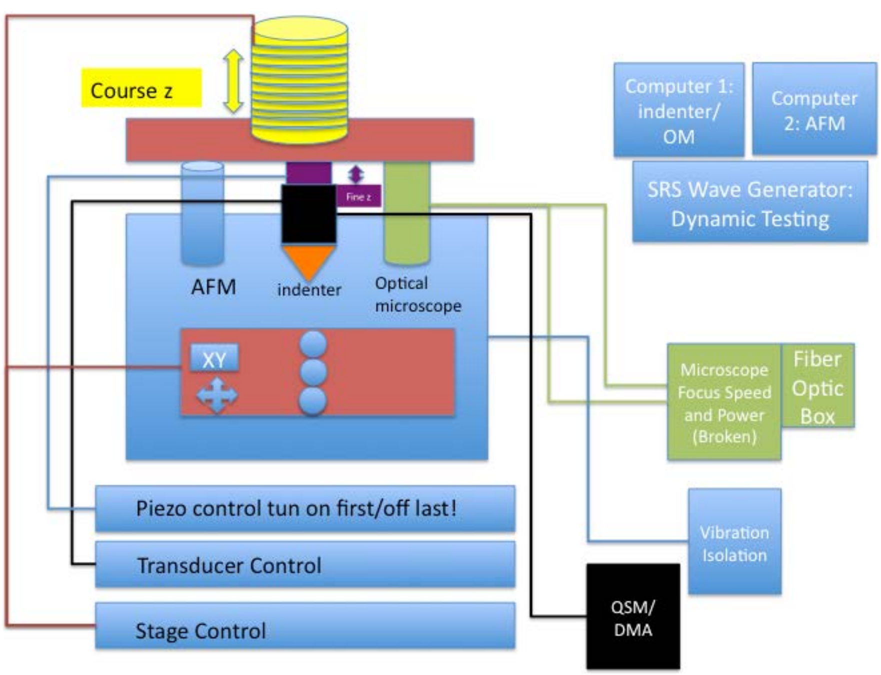
Optical Camera System: A top-down camera is mounted on the right of the piezo scanner. The working distance of the camera is approximately 28 mm. The function of the camera is to allow for optically-driven nanoindentation. The fiber optic illumination box outside of the system controls the intensity of the light supplied to the camera. **Always turn the light off after leaving the tool and during an extended measurement.**

AFM Tip: To the left of the piezo scanner is a mount for an AFM tip for AFM scans. Users typically do not use this function, but it is important to inform the software that there is an AFM head present when starting the software to avoid accidentally crashing the head into the sample or stage.

Motorized stage: As described above, the nanoindenter has a motorized stage used for ‘coarse’ alignment of the sample to the tip. The motors actually have a very high resolution (50 nm in XY direction and 13 nm in Z direction), but the piezo scanner is used to achieve ‘fine’ alignment. The motorized stage controls X, Y, and Z motion. There are three locations on the stage where embedded magnets are used to hold samples. In the current setup, two of the slots are permanently allocated to calibration samples and the third is dedicated for user samples. **Do not remove the calibration samples without first contacting a superuser.**

Vibration Isolation and Acoustic Enclosure: The entire system is mounted onto a granite base, controlled with a dedicated vibration isolation platform and controller, and enclosed in an acoustic enclosure. All of these components help to minimize noise in measurements for overall improved accuracy. **It is worthwhile to note that a pressure difference between the hallway and the room housing the nanoindenter will cause the system to experience an observable vibration when the door is opened. To combat this potential issue, most users leave the door to the room open during the entirety of their experiment.**

f. Tool Schematic Diagrams



II. Theory of Operation and Experimental Setup

The following section describes the theory behind the nanoindentation test and the practical considerations taken for standard tool operation. For more information on the theory behind the test, please refer to the appendix.

a. Schematic Test

In a standard nanoindentation test, a diamond tip with known elastic modulus and poisson ratio is brought into contact with a surface of interest. A force is applied causing the tip to begin to indent the sample and the displacement is measured as a function of this applied force. The parameters in a typical experiment are often: maximum applied force, loading rate, hold time, and unloading rate.

In order to extract materials properties of the sample in question, the machine must be properly calibrated to eliminate the effects of all of the components involved in the measurement other than the sample itself. Two main calibrations are necessary: tip area function and machine compliance, to be described shortly.

Ultimately, the sample's *reduced modulus, E_r,* is measured by extracting fitting data extracted along the unloading portion of the applied load. In this segment, the force applied to the sample decreases from the maximum force down to zero applied force. During unloading, the measured displacement as a function of applied force can be converted into a stiffness value:

$$S = \frac{dP}{dh}$$

where S is stiffness, P is pressure, and h is contact depth. Pressure can be determined by relating the applied force to the contact area using a tip area function that corresponds to the diamond tip geometry. E_r is directly proportional to this stiffness value:

$$E_r = k\frac{S}{\sqrt{A_h}}$$

where k is a constant related to the tip geometry and A(h) is the pre-determined tip area function.

In addition to measuring the reduced modulus, the nanoindentation test can extract the material's hardness using the following simple relationship:

$$H = \frac{P_{\max}}{A_r}$$

where P_{max} is the maximum applied pressure and A_r is the residual indentation area.

i. *Machine Compliance*

Machine compliance refers to the component of measured stiffness that is due to the transducer/tip component of the system. For a given transducer/tip configuration, the compliance should be a constant value across all measurements. Therefore, if the machine compliance is known, it can be subtracted from the total measured compliance to determine the actual sample stiffness:

$$\left(\frac{dP}{dh}\right)^{-1} = C_T = C_M + C_S$$

where C_T is total compliance, C_M is machine compliance, and C_S is sample compliance.

ii. *Tip Area Function*

As previously described, the extracted reduced modulus as well as the extracted material hardness depend on the contact area between the tip and sample, which itself depends upon tip geometry. This relationship is typically a very high order polynomial function, but to first order for a Berkovitch tip:

$$A = 24.5h_c$$

where h_c is the final contact depth. This final contact depth is determined from the displacement data measured using the Berkovitch tip. The tip area function may change over time, and must be empirically determined in order to accurately extract modulus and hardness values.

b. Auto Zero (Note: the fine knob does not work well)

The nanoindenter transducer controller is the electronic component that determines the compliance of the measurement and ultimately the compliance of the sample of interest. However, before taking any critical measurements, the offset value of the tool should be tared (set to zero). In order to do this, simply press the zero button and then adjust the **coarse** dial until the measured value reads +/- 0.01 mg.

c. Workspace and Sample Boundaries ("Sample Safety Zones")

To accurately and safely perform measurements, users must work within a defined *workspace*. The workspace includes all relevant constants for the system setup such as machine compliance, tip area function, transducer calibration data, load control functions, methods, and *sample boundaries* (also known as, "sample safety zones"). Sample boundaries are a critical subset to the workspace. In order to perform a test and bring the tip into contact with the sample, the user must define a sample boundary beforehand. This boundary is generally defined as the field of view of the microscope camera when performing alignments to the substrate. The tool will take note of the range of X and Y coordinates of this boundary and allow the user to bring the tip into contact with the sample **only within the limits of the sample boundary.** Therefore, if a user wants to test multiple locations on the same sample, typically this requires multiple sample boundaries to be defined. In order to bring the tip into contact with the sample, users must perform what is called a *quick approach*, which is a routine where the tip rapidly approaches the sample surface until it comes into contact with the surface. The Z height recorded when this bumping takes place is used to set the safety height, discussed later. When a workspace is saved, all of these sample boundary coordinates and safety heights are saved and a user can pull this data back up to perform additional tests at a later date. **Since no user realistically sets their samples in exactly the same place between two experiments, the policy for this tool is to ALWAYS delete your workspace when**

you have completed and experiment. Also, you can right click in the sample safety zone to navigate within the boundary.

d. Fly Height and Safety Height

When using the nanoindenter, the height offset between the top surface of the sample and the bottom point of the tip is important to note. There are two height values that get programmed into the workspace.

The fly height refers to the distance between the tip and surface when the sample moves out of one sample boundary into another sample boundary. This height is generally a very large distance from the sample surface in order to prevent any potential crashes of the tip with sample due to non-planarity of the sample.

The safety height is the height difference between the tip and sample surface within a sample boundary *after a quick approach has been performed.* The quick approach determines the Z height of the sample surface and the safety height is set as being 100 µm above this absolute Z height. (the figure below, taken from a Hysitron Manual, is incorrect) During an experiment, when subsequent indents are requested within the same sample boundary, the tip will move at this set safety height rather than the fly height during X and Y stage movements.

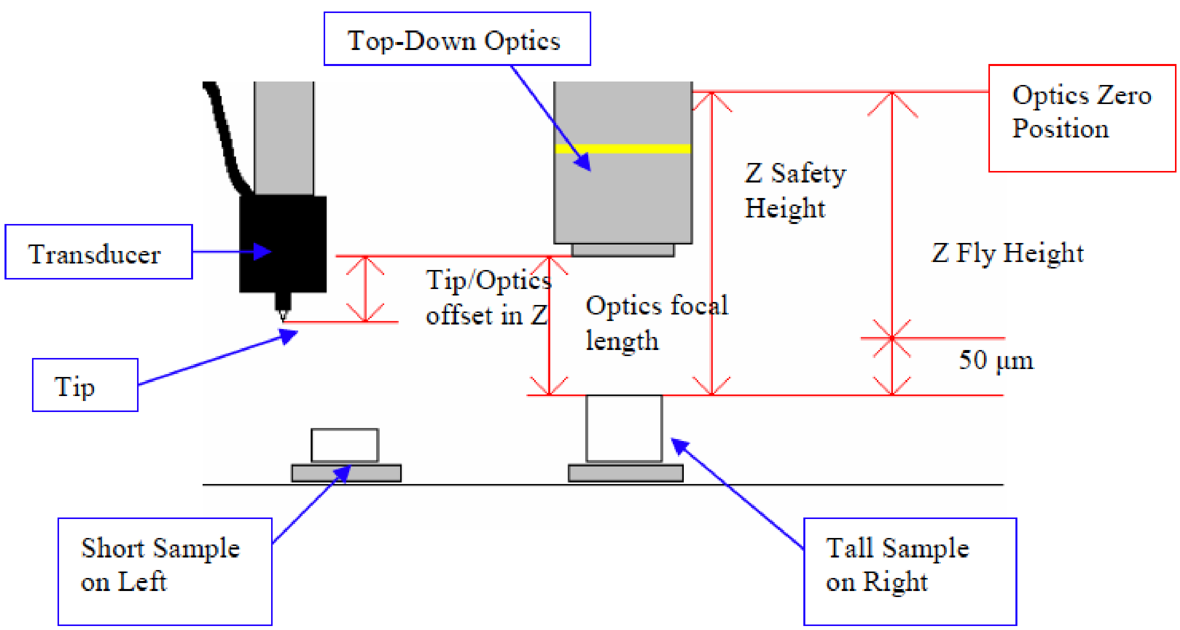


Figure 3.6: Z Fly Height

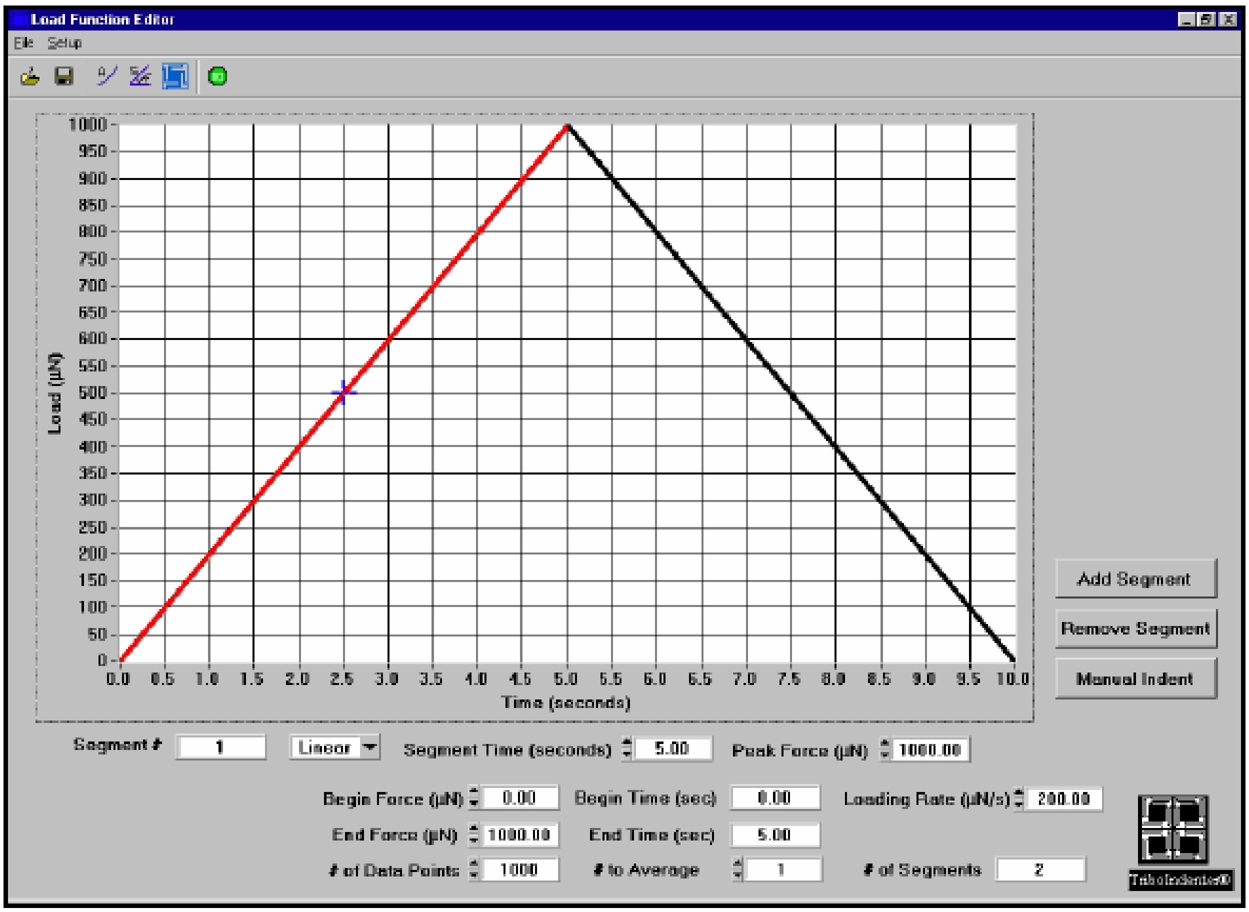
e. Load Functions

Load functions describe the actual indentation test to perform. All load functions are specified as desired load as a function of time. Users can define either open-loop or load-control functions as further described below:

i. *Open-Loop Load Functions*

Open-loop load functions are functions that do not rely on feedback systems to continually drive the required amount of force. The only practical application of open-loop load functions is when performing air indent transducer calibration.

You can perform triangle or trapezoidal load functions (holding), alter time, peak force and loading rate as seen in the Load Function Editor below. This is the load function used by the optics-tip offset calibration.



ii. *Load-control Load Functions:*

Load-control load functions rely on force measurement feedback from the transducer controller to continuously adjust the applied force to match the specified load function. Load-control load functions are the functions that all "indent from optics" indents use.

iii. *Displacement – control load functions*

The Hyistron Triboindenter is truly a load-control machine. The true feedback is the load, so the displacement control is not truly displacement control: the loading curves can come out very shaky depending on the gains used.

III. Software Tutorial

This software tutorial will introduce you to the TriboScan software and provide some fundamental information about how to operate and navigate the software to perform your indentations. There are many advanced indentation techniques not explicitly explained in this section of the manual, but you can refer to the TriboScan manual if you want further reference. The following guides should be sufficient for day-to-day operation. Below is the TriboScan Toolbar that allows you to navigate to different function windows outlined below

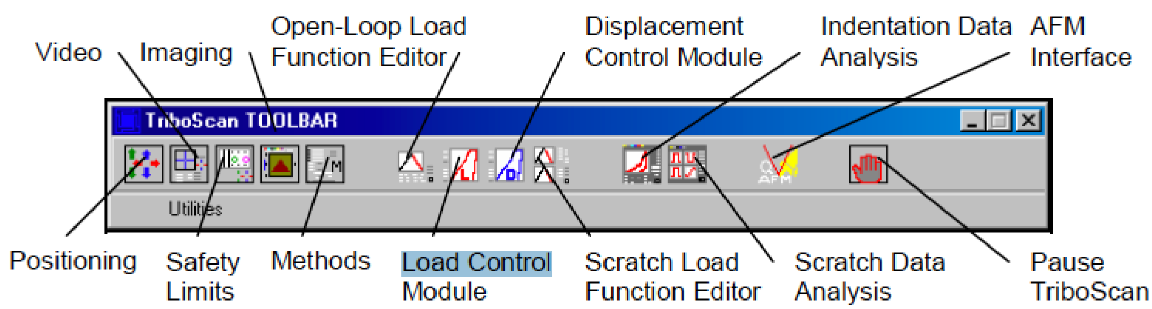


Figure 3.1: TriboScan Toolbar

a. Stage Movement

The Positioning screen allows you to move the stage in the X and Y direction as well as to move the tip toward/away from the stage in the Z direction. There is an option to change the resolution of movement within this screen by selecting coarse, medium, fine, and ultra fine. For most common use, medium and fine resolution are sufficient to position the stage. The current position of the stage is always listed on the bottom left of the screen.

Important functions available to users from within this screen are:

Setup > Calibrate Optics (to be used to calibrate optics if they are offset).

b. Safety Limits

This segment will describe the meaning of safety limits and explain how to set up sample boundaries using the Safety Limits screen.

c. Video Mode

This segment will describe how to use the Video screen to navigate to your sample location.

d. Load Function Setup

This segment will describe the Open-Loop and Load-Control Load Function screens.

e. Data Analysis

This segment will describe the features located in the Data Analysis screen including the Plot Multiple Curves and Multiple Curve Analysis functions.

f. Automated Methods

OVERVIEW

Once all of the safety zones and the probe tip / optics offset have been calibrated, it is possible to move to different locations on samples and save these locations in the software. The TriboIndenter is an optics driven instrument, so all of the movements and locations are stored relative to the optics locations that the user sees in the video window. Sample locations may be stored in three different groups, including Patterns, Position Groups, and Safety Limits.

If you want to make a grid of indents, or make one or two indents in predefined locations (site specific) , you can automate the indentation process. The type of automation you are wishing to perform depends on your sample. You can

1. Set up arbitrary geometric "PATTERNS" (circle array, grid array). Note the direction the indentation occurs (typically serpentine) in case you want to correlate the indent number with the location. These patterns can be placed at a specific location ("origin" specified in absolute global coordinates.) You can decide whether this origin location will serve as the start point, end point, geometric center, upper left position, etc of your array. The coordinates of the indents themselves in the pattern will be defined by local, non absolute coordinates, in reference to the origin position you specified.
2. Save specific locations in absolute global coordinates called "POSITION GROUPS"

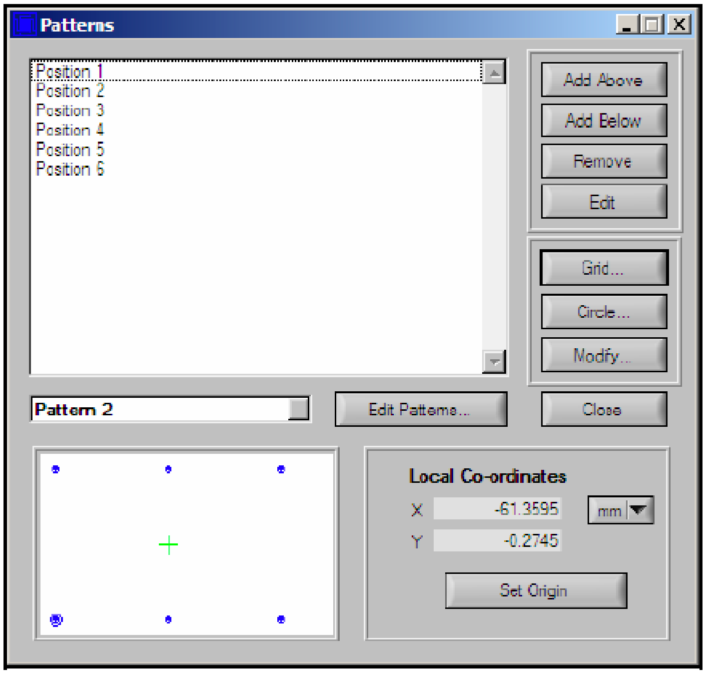
HERE IS WHERE IT GETS CONFUSING: you can tell the method editor to perform a "PATTERN" of indents at particular "POSITION" in a particular "POSITION GROUP" (eg. perform pattern "grid1" at "position1" in the position group "silicon")

You can tell the method editor to perform patterns at all the positions in the position group, with the understanding that those positions are correlated with the patterns as the origin for that pattern (so the pattern will be spatially located around that position accordingly, however you defined the origin for the pattern in the "pattern" editor).

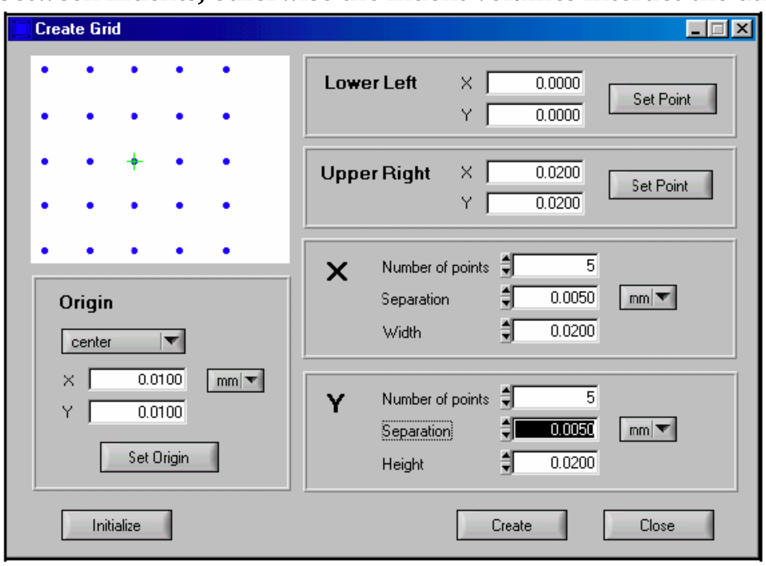
Lastly, if you have some site specific locations, you can save each of those locations either in one position group (if it's in the same sample boundary, eg, all at the SAME HEIGHT) or in different position groups. Sometimes you will have to define different "samples" on your sample even if you have one "contiguous" sample, because the sample itself is too "hilly.".Actually you can have positions in one position group on many samples, but this is questionable…

PATTERNS:

1. Patterns are different from Position Groups in that the locations of each of the indents in the pattern are defined relative to a local zero, rather than an absolute stage coordinate. A predefined grid or circle will be set with positions relative to the green cross.
2. POSITIONING WINDOW -> FILE -> PATTERNS or METHOD EDITOR -> PATTERNS! This will open up the Pattern Editing window (below)



3. To make a PREDEFINED PATTERN: click "Grid" or "Circle" depending on the type of array you want (below). **If doing compliance testing on quartz: 250-100um separation between indents, otherwise the indent volumes interact the data will be wrong.**



4. Number of indents and separation of points can be set in the grid editor.
5. Set "origin" by click on the origin button OR you can manually enter it in. ORIGIN IS GIVEN BY GREEN CROSS, and if you decide to make this pattern at a specific location ("position"), that position will be the green cross position and the other indents will fall around it.
6. Arbitrary geometries: you do not have to use the predefined patterns of "Grid" and "Circle/Ellipse" Simply move the stage, enter in the origin, then move the stage for and click "add above" or "add below" for an arbitrary pattern. Save location is not the absolute position, but rather the distance from the origin to the newly saved point. BECAUSE PATTERNS IS IN LOCAL COORDINATES. The Positions in the pattern can also be added

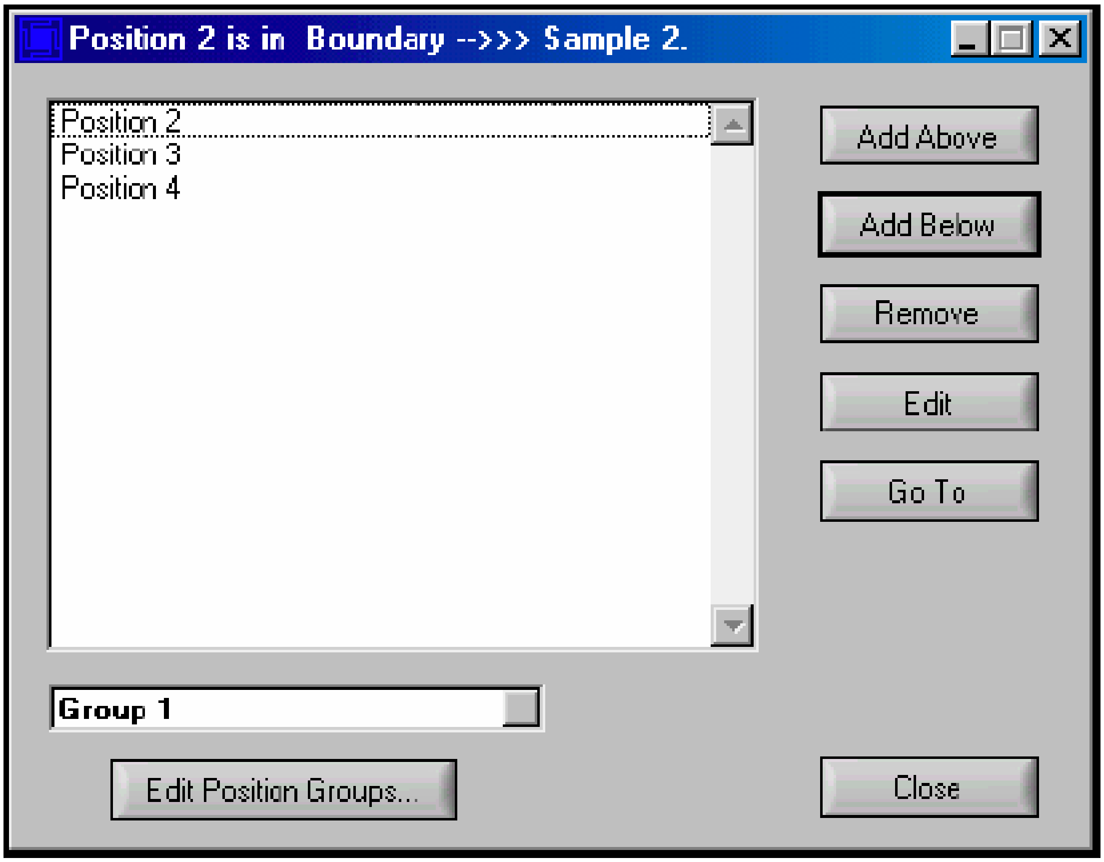
manually in the pattern editor (double click location in the list, or select the location and click edit to change name and position of the location.) So if you want a triangle array, or something similar, this can be accomplished this way.

7. Actually Press "Create".

POSITIONS GROUPS:

Position Groups are groups of specific locations set up by the user. Position groups differ from Patterns in that the locations in the groups are defined as absolute, rather than local, stage coordinates. In automated indentation experiments, patterns are placed at each of the locations in a position group. Position Groups may be a single location, several locations in a sample safety zone, or locations on several different samples.

1. POSITIONING WINDOW -> FILE -> POSITION or METHOD EDITOR -> POSITIONS! This will open up the Position Editing window (below)

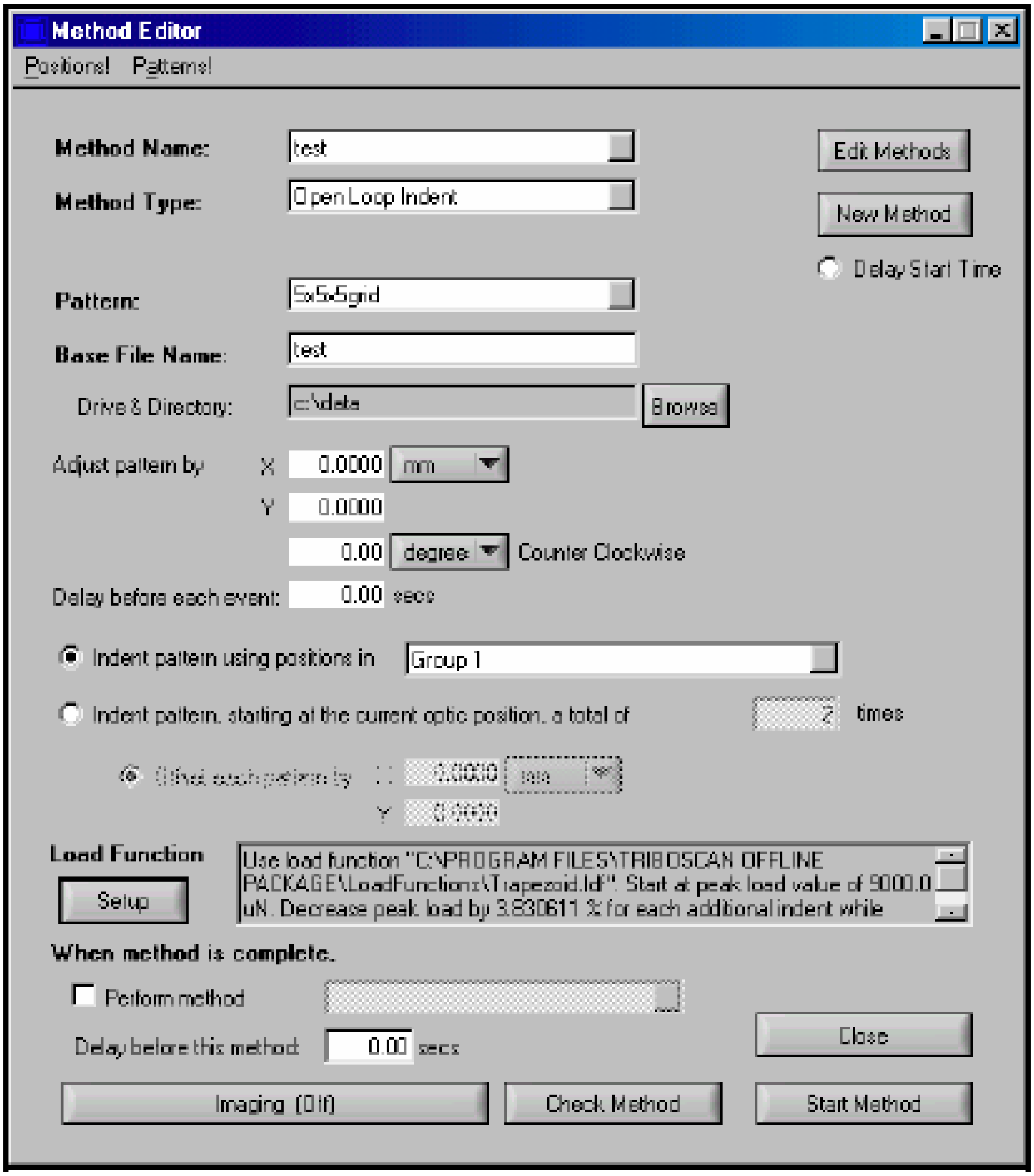


2. You can add positions by moving the stage and clicking "add above" or "add below". Positions can also be added manually in absolute coordinates by clicking "Edit" on the particular position highlighted. Note it says "Position 2 is in the Boundary "Sample 2". You can press "go To" for the stage to "go to" that position.
3. If you wan to great a new Position Group, click on "Edit Position Groups".

AUTOMATED METHOD EDITOR

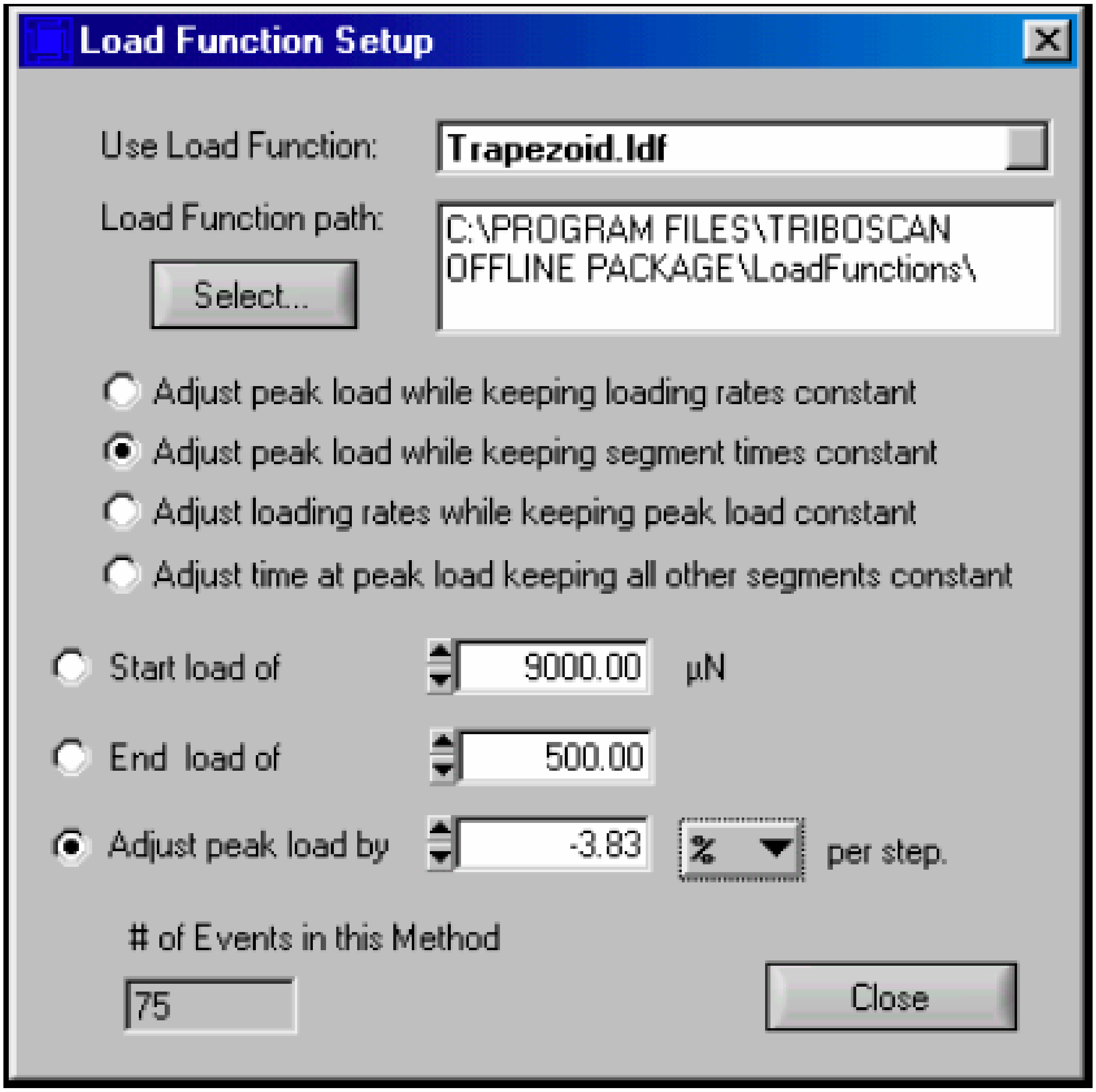
The Method Editor is the control center for automated experiments. It is a simple, but powerful tool for setting up standard or new experiments. It utilizes Patterns and Position Groups to set up a series of tests.

1. POSITIONING WINDOW -> METHODS -> NEW METHOD



2. Pick the "Method Type": Indent, Scratch, or Wear Test.
   a. *Troubleshooting note: if you pick Open Loop Indent, then the only files that can be accessed when pressing "setup" under "Load Function" will be ldf files, you won't even be able to access any load control loops. So make sure you pick the right one you want!*
3. Select the Pattern you want. This should already be saved as a pattern if you have made a pattern in the patterns editor: can be accessed by clicking on the "PATTERNS!" menu in the window
4. Base file name: if you're base file name is "silicon" then your data for each indent will be a file named "silicon_##.hys"

5. You can save all the indent file data in to the directory of your choosing: please choose PROGRAM FILES -> HYSITRON TRIBOINDENTER -> USER DATA -> YOUR FILE.
6. Set the position group: so now you can choose the absolute coordinates (that you saved earlier) where you will end up indenting your pattern. If you don't have multiple positions, and simply want to indent where the optical microscope is focused on currently, then you can select "Indent pattern starting at the current optic position"



7. A method will use ONE LOADING FUNCTION and can adjust ONE parameter for each indent.
   a. First pick your saved basic loading function (you can end up changing the max load, segment times, loading rates etc, but specify the parameters you think you want)
   b. It can adjust the peak load (so you can have a pattern where the max load for each indent decreases or increases) while keeping loading rates constant (so the loading rate you specified in the initial load function will use that rate) so if you go from 10000uN to 1000uN and the rate is 1000uN/sec, your first indent will take 10 seconds to load and the last will take 1 second to load (probably not desirable)

c. Adjust peak loads while keeping segment times constant (more desirable, each indent will take the same amount of time to load, much steadier data, less errors)
d. Adjust loading rates for each indent (keeping max load the same for all indents)
e. Adjust time at peak load keeping all other variables constant
f. IF YOU JUST WANT THE SAME MAX LOAD: CHOOSE ADJUST PEAK LOAD WHILE KEEPING SEGMENT TIMES CONSTANT, AND CLICK ADJUST PIEAK LOAD BY 0UN OR 0% PER STEP.
g. If you want to change more than one of these variables, another method should be used per variable changed

8. Click “check method”. Box will pop up telling you to save your workspace if you haven’t and it will check
   a. If any indents in the patterns fall outside sample safety zones
   b. Any loads are outside the capacity of the tranducer
   c. It will print out a method summary: you can save as a text if you want
   d. Preflight will have the triboindenter move the stage under the tip for each position specified without actually performing indents
9. Click start method.

IV. Standard Machine Operation

The following section describes the standard operation procedure for performing a single indent.

a. Power On

When arriving to the machine, note that the following tool conditions are met:

Stage Controller	→	ON
Transducer Controller	→	OFF
Piezo Controller	→	OFF
Software	→	CLOSED
Camera Light Source	→	OFF

If any of the above conditions are not met, please note in the log book.

When beginning to operate the tool, perform the following steps:

1. Turn on the Piezo Controller
2. Turn on the Transducer Controller and **zero the scale**
    a. **Please: change the display gain to see the tip weight before taring. Note the tip weight in the Logbook (~268mg). Then change the display gain back to 0.000. THE WEIGHT OF THE TIP ON THE TRANSDUCER MUST BE NEAR ZERO BEFORE OPENING THE SOFTWARE.**
3. Start the TriboScan software located on the desktop
    a. Click Cancel when prompted to open in Analysis Mode Only
    b. Click QSM when prompted about Quasi-static mode
    c. Confirm that the Data Acquisition Box (blue box on desk) is set to QSM
    d. Click AFM Tip Present when prompted about AFM head
    e. Follow the Rest of the On Screen Instructions
    f. AUTO ZERO THE LOAD AGAIN BEFORE YOU BEGIN ANY TESTING. ESPECIALLY IF YOU FORGOT TO FOLLOW STEP TWO.
4. Turn on the Camera Light Source

b. Air Indent Calibration

At this time, the system can now be calibrated to confirm normal operating conditions. First, perform and air indent calibration by doing the following:

1. Open the Open-Loop Load Editor
2. Program an open-loop load for a 600 µN, 2-segment load with 10 second loading and unloading times (total time equals 20 seconds).
    a. ALTERNATIVELY: open Triboscan->Calibrations->Z-cal
3. Set the Displacement Gain on the transducer controller to 100 (This value should be used for ALL indents that are anticipated to be greater than 100 nm in depth.)
4. Open the Transducer Constants screen in the software (by clicking the blue symbol in the open load window) and adjust the displacement gain to 100
5. Set the Microscope Feedback Gain on the transducer controller to 100
    a. Open the Positioning Screen
    b. Naviagate to Setup > Piezo Control

   c. Edit the Microscope Feedback Gain value
6. Navigate to Open Loop Load function
7. Select Setup > Advanced Z-Axis Calibration
8. Set the bias offset to 0.2 V
9. Check the Transducer Zero-Volt ESF value.
   a. If the value is reasonably different (greater than 10% from the transducer datasheet value) report this in the log book and reset the value to the datasheet value. (It should be ~0.1318 uN/V^2 for the high load transducer and 0.02513uN/V^2 for the Low Load)
   b. If the value is reasonably similar to the datasheet value, do not adjust it before the test is performed.
10. Set the Transducer Zero-Volt Gap to ~38um for the High Load Transducer (50mN max) [80microns for the Low Load transducer, 10mN Max]
11. Click the Calibrate Transducer button to perform the air indent calibration and follow the onscreen instructions (the Transducer constants window will pop again. Double check that the blue values are correct initial guesses)
12. When complete, the data present should look fairly noisy and centered on zero. (F-D curve) If this is not the case, attempt to perform the air indent again. If you are still not seeing the expected results, note this in the log book and contact a super user before continuing.
13. Return the Microscope Feedback gain to 1000 on the transducer controller
   a. Open the Positioning Screen
   b. Naviagate to Setup > Piezo Control
   c. Edit the Microscope Feedback Gain value
14. Mark down ESF/Max F and Plate spacings on the logbook (blue symbol -> blue values)

c. Aluminum Optics Calibration and Tip Cleaning

The next step is to perform a quick indentation into the aluminum calibration sample. The purpose of this test is two-fold. First, if the indent observed after the calibration is complete is roughly located where the crosshairs overlaid on the image are located, you have confirmed that the optics offset has not changed. Second, a quick indentation into aluminum is often used to clean the tip of any potential dust or other contamination that may lead to an inaccurate test.

1. Turn on the video screen
   a. Ensure the light source power is on and the intensity knob is not turned too far down (you should see some light bleeding out from the box).
   b. Open the Video screen in the TriboScan software
   c. Set the white level and the black level both to 30
2. Position the camera above the aluminum sample
   a. Open the Safety Limits screen in the TriboScan software
   b. Disable the XY Safety Limits and Z Safety Limits (At this point you are at risk of crashing the tip into the sample/stage without the software preventing you from moving the motors outside of safe operating ranges. Be careful to always watch the tip location while moving fin x, y, z. Move the mouse physically to on top of the computer so you may watch the screen and stage at the same time).
   c. Open the Positioning screen in the TriboScan software
   d. Move the camera above the aluminum sample by positioning the light from the camera on top of the aluminum. Change the resolution of the stage movement to Medium if the stage is moving too slowly.

3. Focus on the aluminum sample surface
    a. Move the camera down toward the sample using the Z-axis control. Be careful to watch the tip location while doing this.
    b. Verify you are above the sample by simultaneously checking the Video screen to observe the image.
    c. When focused, the position of the Z height of the tip is often roughly 25.88 mm. (for the green indenter position).
4. Create a sample boundary and prepare for indent
    a. Re-navigate to the Safety Limits screen and click Create Boundary
    b. Re-enable the XY Safety Limits and the Z Safety Limits
    c. Save your workspace.
    d. Perform a quick approach by clicking the Quick Approach button on the right side of the panel. Note that the Last Contact and Safety Height value listed for this particular sample boundary will update after the quick approach is complete. They should 100 µm different. **Before every quick approach you must re zero the transducer because it is approaching the sample: it uses force feedback to determine where the surface of the specimen is, and should start with zero force on it.**
    e. Navigate to Load-Control Screen
    f. Load pre-configured load-control file from:
    Parameters menu: UserData\Calibrations\aluminum\aluminum.fbp
5. Perform and Analyze indent
    a. From Load Control screen select Indent From Optics button (From Load Control screen select Indent From Optics button and follow on-screen instructions). **(always as a check: did you quick approach and re-zero the transducer, and lastly, displacement and microscope gains match?)**
    b. When indent is complete, make sure to save data to: User Data\Calibrations\aluminum\
        i. Make sure to make a new sub-directory with the current date (yyyy-mm-dd)
        ii. Save the file with your initials
    c. After indent, the tool should automatically return the camera to a position immediately above the intended indent location
    d. Confirm that a visible indent is present within a reasonable distance of the crosshairs
    e. Confirm successful test by marking **log book**

d. Quartz Compliance/Tip Area Function Test

The purpose of this test is to confirm that the previously loaded tip area function and machine compliance calibration yield results are still valid for future tests. To do this, an indentation is performed on the quartz sample and the extracted E_r and hardness values are recorded/compared to expected results. If the values fall outside of expected tolerance levels, then the machine needs to be re-calibrated before the results can be accurately analyzed. Most likely the machine compliance has gone out of calibration. It is possible to continue to perform your tests and then re-analyze the data in the future with the updated compliance and tip area function values, though you acknowledge the potential for inaccurate data extraction by electing to re-analyze your data in the future.

1. Position the camera above the quartz sample
    a. **MOVE MOUSE PHYSICALLY TO A LOCATION WHERE YOU CAN WATCH THE STAGE AND VIDEO SCREEN CONCURRENTLY**

b. Open the Safety Limits screen in the TriboScan software
c. Disable the XY Safety Limits and Z Safety Limits (At this point you are at risk of crashing the tip into the sample/stage without the software preventing you from moving the motors outside of safe operating ranges. Be careful to always watch the tip location while moving forward).
d. Open the Positioning screen in the TriboScan software
e. Move the camera above the quartz sample by positioning the light from the camera on top of the quartz. Change the resolution of the stage movement to Medium if the stage is moving too slowly.

2. Focus on the quartz sample surface
   a. Move the camera down toward the sample using the Z-axis control. Be careful to watch the tip location while doing this.
   b. Verify you are above the sample by simultaneously checking the Video screen to observe the image.
   c. When focused, the position of the Z height is often roughly 28 mm. If you are having trouble focusing, try to focus on the edge of the sample or go to the center and focus out until you see the black dots
3. Create a sample boundary and prepare for indent
   a. Re-navigate to the Safety Limits screen and click Create Boundary
   b. Re-enable the XY Safety Limits and the Z Safety Limits
   c. Save your workspace.
   d. Perform a quick approach by clicking the Quick Approach button on the right side of the panel. Note that the Last Contact and Safety Height value listed for this particular sample boundary will update after the quick approach is complete. They should 100 µm different. (before quick approach, remember to re-zero the transducer control)
   e. Navigate to Load-Control Screen
   f. Load pre-configured load-control file from: Calibrations\quartz\quartz.fbp: Check 5000uN max at 1000uN/s load and unload, with 2 second hold
4. Perform and Analyze indent
   a. From Load Control screen select “Single Indent From Optics” button **(always as a check: did you quick approach and re-zero the transducer, and lastly, displacement and microscope gains match?)**
   b. When indent is complete, make sure to save data to: User Data\Calibrations\quartz\
      i. Make sure to make a new sub-directory with the current date (yyyy-mm-dd)
      ii. Save the file with your initials
   c. After indent, the tool should automatically return the camera to a position immediately above the intended indent location and the Data Analysis screen should automatically open. If the Data Analysis screen does not open, open it and load the data from the indent
   d. Ensure the unloading segment is listed as segment #3
   e. Ensure the upper and lower fit values are 95% and 20%, respectively.
   f. Click the Execute Fit button
   g. A green line should be overlaid along the unloading portion of the curve and the E_r and Hardness fields should populate with the extracted values.
   h. Record the values in the log book.
   i. If the values fall within expected limits, proceed with your experiment. If they are outside of the range, contact the super user immediately and proceed at your own risk.

e. Experiment

At this point you are ready to perform your own experiment. You can perform single indents from the Load Control screen or run methods to automate multiple measurements. You will choose whatever technique is most appropriate for the test you wish to complete, but the following steps should be taken before you can start.

1. Load your sample
   a. Your sample should be glued to a magnetic specimen disk and adequately dried before testing
   b. When dry, load your sample onto the third (and only available) magnetic location on the sample stage.
2. Position the camera above the your sample
   a. Open the Safety Limits screen in the TriboScan software
   b. Disable the XY Safety Limits and Z Safety Limits (At this point you are at risk of crashing the tip into the sample/stage without the software preventing you from moving the motors outside of safe operating ranges. Be careful to always watch the tip location while moving forward).
   c. Open the Positioning screen in the TriboScan software
   d. Move the camera above the aluminum sample by positioning the light from the camera on top of the sample. Change the resolution of the stage movement to Medium if the stage is moving too slowly.
3. Focus on the sample surface
   a. Move the camera down toward the sample using the Z-axis control. Be careful to watch the tip location while doing this.
   b. Verify you are above the sample by simultaneously checking the Video screen to observe the image.
   c. When focused, the position of the Z height is often roughly 28 mm.
4. Create a sample boundary and prepare for indent
   a. Re-navigate to the Safety Limits screen and click Create Boundary
   b. Re-enable the XY Safety Limits and the Z Safety Limits
   c. Save your workspace.
   d. Perform a quick approach by clicking the Quick Approach button on the right side of the panel. Note that the Last Contact and Safety Height value listed for this particular sample boundary will update after the quick approach is complete. They should 100 µm different.
5. Perform experiment
   a. Load your method of choice or perform single indentations from optics from within the load control screen.
   b. Make sure to save your date in your User Data folder located in the directory: ???
6. Analyze your data
   a. Open the Data Analysis screen. If performing a method you must wait until all indentations are complete.
   b. Perform analysis on the unloading segment of your data.
   c. Perform multiple curve analysis on multiple curves by selecting the Multiple Curve Analysis button in the toolbar. Selecting this option will prompt you to select the desired curves to analyze from your data folder. You will also be prompted for a place to save the data. The saved data will be a tab-delimited text file with all relevant extracted parameters

listed per curve chosen in the analysis. The output will be a panel with two graphs: Hardness vs. Contact Depth and Reduced Modulus vs. Contact Depth.

d. Plot multiple curves on a single plot by selecting the Plot Multiple Curve button in the toolbar. Selecting this option will prompt you to select the desired curves to analyze from your data folder. The output will be a panel with a single axes with all of your plots shown at once. You can highlight a plot by clicking on it with the mouse or by selecting it in the menu and remove it from the plotted curve set if desired. You can also select the Multiple Curve Analysis button in this panel to perform multiple curve analysis (as described immediately above) on only the curves shown in this plot.

f. Machine Shutdown

After you have completed performing all of your tests and analysis, make sure to save your data and then shut down the tool as follows:

1. Shutdown the Software.
    a. During shutdown the machine should move to the default optics location.
2. Remove your sample from the stage.
3. Delete the workspace you saved in order to perform your calibrations and indentations. (ProgramFiles -> TriboScan -> Workspace -> YOUR FOLDER)
4. Turn off the Camera Light Source.
5. Turn off the Transducer Controller.
6. Turn off the Piezo Controller.
7. Sign the log book.

V. Qualification Procedures

a. Standard Qualification

The standard qualification procedures will grant you access to the tool for use of the 1-D transducer ONLY. The procedure is:

1. Review all of the online course materials (manuals and demonstration videos) to become familiar with the tool
2. Contact a qualified user to set up a time for training. This training must consist of:
   a. Discussion of tool components
   b. Demonstration of complete standard operation
   c. Carefully monitored trainee use of the machine
      Note: The monitored use *may* include use of a trainee's specific sample, but it should not be an exhaustive analysis of the trainee's material. Rather, this should consist of one or two indentations.
3. Schedule additional training sessions, nominally with different users if possible, until you are comfortable using the tool.
4. Contact superuser to inform he/she that you have completed a training session and are prepared to become qualified. Set up a time to demonstrate proper tool use for superuser in order to complete qualification.

b. Advanced Qualifications

Advanced qualifications will permit you to use functions such as the 2-D transducer, dynamic testing, and submerged testing. Due to the complicated nature of these tests, you must complete a few additional qualifications before being permitted to use these advanced functions. Some specifics are listed below. If a function that you wish to use is not detailed below, contact the super user for instructions.

1. Transducer Removal and Calibration Training Requirements
   a. Successful demonstration of 1-D transducer use
   b. Working knowledge of theoretical background for nanoindentation tests as well as tool operation
   c. Contact super user for Transducer Removal and Calibration training and instructions
   d. Perform Compliance Calibration in quartz sample with limits:
      i. 49 Indents (7x7 grid)
      ii. Load control profile with 10 second load, 2 second wait, 10 second unload
      iii. Max force = 27mN
      iv. Min force = 50 µN
      v. Vary force linearly between indents
      vi. Properly analyze data and update settings
   e. Perform Tip Area Function Calibration in quartz sample with limits:
      i. 49 Indents (7x7 grid)
      ii. Load control profile with 10 second load, 2 second wait, 10 second unload
      iii. Max force = 27mN
      iv. Min force = 50 µN
      v. Vary force by approximately 12% (decreasing from max to min) between indents
      vi. Properly analyze data and update settings

2. 2-D Transducer Training Requirements
    a. Successful demonstration of 1-D transducer use
    b. Complete training for transducer removal and calibration
    c. Contact super user for 2-D Transducer training instructions

VI. Appendices

a. Troubleshooting Guidelines/Procedures

i. Timeout error

1. You did not actually focus on the sample surface, (most likely above it), so the Z motion of the indenter piezoelectric is extending, but cannot "feel" the pushback of your sample as it extends. It times out after 7 minutes. Erase your sample, and create the sample again, this time ensuring you are focusing on the sample surface (can be difficult with transparent samples to find the focus
2. You did not perform a QUICK APPROACH
   a. Do I seriously have to remind you again to do a quick approach before any tests????
3. It's possible the tip-optics offset isn't correct and it's not indenting anywhere near the optical location: look to see where the stage is moved when you try to perform the indent. If it's not near where you think it's supposed to be indenting, then the optics-tip offset needs to be recalibrating.
4. Dirty tip or dirty sample. For dirty tip, re-indent aluminum.

ii. Tip feels excessive force

1. **Excessive force continues even with restarting: Did you tare the transducer before turning on the software. Seriously. How many times do I have to ask this?**
2. Are you trying to perform an indent, and before the tip "stepped towards the surface" you had this error? Then you focused the sample when creating the sample boundary BELOW the surface of the sample, and the coarse z motion quickly encountered your sample and the transducer felt the pushback immediately. Delete your sample and refocus correctly and try again. Before you can do this however, you must
   a. Follow the onscreen instructions to rotate the yellow knob clockwise (looking down) which will move the tip away from the surface manually. Then you must home the z axis, enable the motors, and then you can perform your test again
3. Did you receive this error when the stage was moving and you were not even performing an indent?
   a. The door to the acoustic shield is open. Close it and make sure no other doors to the room are being open and closed, the gust of air and pressure difference is enough for the transducer to feel.
4. Did you receive this error as the tip was "stepping toward" (fine z, piezo extension motion) the sample?
   a. Loading rate might be too high.

iii. Shaky data

1. Did the ESF value, plate spacing and max force match the transducer constants sheets?
2. Do the displacement gain and microscope feedback values match between hardware and software IF THEY DON'T, THE INDENTER IS CONFUSED, AND YOU HAVE TO RESTART THE SOFTWARE AND THE HARDWARE FOR THE CONFUSION TO END.
3. Try different loading rates
4. Dirty tip/Dirty sample/dirt in the transducer

iv. Frozen Drift Monitor

1. Click on it.

v. Hysteresis in air indent calibration curve. Also Force-Displacement curve looks funny for air indent

1. Transducer is dirty. It needs to be cleaned. This can typically be accomplished by taking the tip off the transducer and then reinserting both. This alone can clean any debris that is caught.

vi. **How should I test my sample? What's the best way to mount it? What the best range of loading?**

1. THIN FILMS RULE OF THUMB: don't indent too shallow (surface effects) and don' indent too deep (substrate effects).
2. Read the relevant literature in your field!
3. Contact the hysitron technicians
4. The only papers we can help you with are for the DATA ANALYSIS

b. Nanoindentation Literature References

i. Doerner, M.F. and Nix, W.D., *A method for interpreting the data from depth-sensing indentation instruments*, Journal of Materials Research, Vol. 1, No. 4, p. 601 (1986).

ii. Oliver, W.C. and Pharr, G.M., *An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments,* Journal of Materials Research, Vol. 7, No. 6, 1564 (1992).

c. Hysitron User Manual

i. Hyistron Manual is appended to this manual